```
data_lach4

_publ_section_comment
;
The allyl (C3H5) group on the top of the cluster is disordered by
symmetry over three sites.
Squeeze was carried out on the structure, to make allowance for
disordered solvent molecules.
;

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             (NBu4)3H[SiW9O34(tBuSiO)3(SiC3H5)]
_chemical_melting_point           ?
_chemical_formula_moiety          ?
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 'C62 H138 N3 O37 P Si4 W9'
_chemical_formula_weight          3315.73

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 _atom_type_scat_dispersion_imag
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 'C'  'C'   0.0181   0.0091
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0492   0.0322
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Si'  'Si'   0.2541   0.3302
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'W'  'W'  -5.4734   5.5774
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.2955   0.4335
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0311   0.0180
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            Trigonal
_symmetry_space_group_name_H-M    'R 3 c'

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-y, x-y, z'
 '-y, -x, z+1/2'
 '-x+y, -x, z'
 'x, x-y, z+1/2'
 '-x+y, y, z+1/2'
 'x+2/3, y+1/3, z+1/3'
 '-y+2/3, x-y+1/3, z+1/3'
 '-y+2/3, -x+1/3, z+5/6'
 '-x+y+2/3, -x+1/3, z+1/3'
 'x+2/3, x-y+1/3, z+5/6'
 '-x+y+2/3, y+1/3, z+5/6'
 'x+1/3, y+2/3, z+2/3'
 '-y+1/3, x-y+2/3, z+2/3'
 '-y+1/3, -x+2/3, z+7/6'
 '-x+y+1/3, -x+2/3, z+2/3'
 'x+1/3, x-y+2/3, z+7/6'
```

```
 '-x+y+1/3, y+2/3, z+7/6'

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_cell_length_b                    22.4981(4)
_cell_length_c                    36.7371(11)
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_cell_angle_beta                  90.00
_cell_angle_gamma                 120.00
_cell_volume                      16103.7(6)
_cell_formula_units_Z             6
_cell_measurement_temperature     296(2)
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_exptl_crystal_density_diffrn     2.051
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              9384
_exptl_absorpt_coefficient_mu     18.439
_exptl_absorpt_correction_type    Multi-scan
_exptl_absorpt_correction_T_min   0.1197
_exptl_absorpt_correction_T_max   0.4042
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_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       296(2)
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_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'Bruker Smart 6000 CCD'
_diffrn_measurement_method        'CCD scans'
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_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             31955
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_diffrn_reflns_av_sigmaI/netI     0.0629
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_diffrn_reflns_limit_k_max        26
_diffrn_reflns_limit_l_min        -43
_diffrn_reflns_limit_l_max        44
_diffrn_reflns_theta_min          3.31
_diffrn_reflns_theta_max          70.21
_reflns_number_total              6308
_reflns_number_gt                 4227
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        SMART
_computing_cell_refinement        SAINTPLUS
_computing_data_reduction         SAINTPLUS
_computing_structure_solution     SHELXTL
```

```
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor
wR and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold expression
of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0390P)^2^+230.6009P] where P=(Fo^2^+2Fc^2
^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    -0.01(2)
_refine_ls_number_reflns          6308
_refine_ls_number_parameters      355
_refine_ls_number_restraints      178
_refine_ls_R_factor_all           0.0811
_refine_ls_R_factor_gt            0.0466
_refine_ls_wR_factor_ref          0.1184
_refine_ls_wR_factor_gt           0.0985
_refine_ls_goodness_of_fit_ref    1.102
_refine_ls_restrained_S_all       1.096
_refine_ls_shift/su_max           0.001
_refine_ls_shift/su_mean          0.000

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 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
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 _atom_site_disorder_group
C1 C 0.7961(13) 0.5557(13) 0.7347(6) 0.110(6) Uani 1 1 d U . .
C2 C 0.7473(18) 0.5549(18) 0.7094(9) 0.203(14) Uani 1 1 d U . .
H2A H 0.7708 0.5807 0.6881 0.304 Uiso 1 1 calc R . .
H2B H 0.7229 0.5750 0.7206 0.304 Uiso 1 1 calc R . .
H2C H 0.7154 0.5084 0.7026 0.304 Uiso 1 1 calc R . .
C3 C 0.8328(18) 0.6230(14) 0.7535(8) 0.186(12) Uani 1 1 d U . .
```

H3A H 0.8797 0.6475 0.7454 0.280 Uiso 1 1 calc R . .
H3B H 0.8315 0.6158 0.7793 0.280 Uiso 1 1 calc R . .
H3C H 0.8110 0.6493 0.7478 0.280 Uiso 1 1 calc R . .
C4 C 0.8531(18) 0.5536(17) 0.7151(9) 0.192(12) Uani 1 1 d U . .
H4A H 0.8476 0.5562 0.6894 0.289 Uiso 1 1 calc R . .
H4B H 0.8522 0.5115 0.7208 0.289 Uiso 1 1 calc R . .
H4C H 0.8962 0.5918 0.7227 0.289 Uiso 1 1 calc R . .
C5 C 0.6667 0.3333 0.6861(9) 0.130(15) Uani 1 3 d SDU . .
H5 H 0.6775 0.3739 0.6741 0.156 Uiso 0.33 1 calc PR . .
C6 C 0.652(5) 0.280(4) 0.667(2) 0.18(4) Uiso 0.33 1 d PDU . .
H6A H 0.6413 0.2383 0.6779 0.216 Uiso 0.33 1 calc PR . .
H6B H 0.6530 0.2823 0.6413 0.216 Uiso 0.33 1 calc PR . .
C7 C 0.5502(13) 0.5245(13) 0.7752(8) 0.124(6) Uani 1 1 d U . .
H7A H 0.5274 0.4780 0.7845 0.148 Uiso 1 1 calc R . .
H7B H 0.5989 0.5443 0.7801 0.148 Uiso 1 1 calc R . .
C8 C 0.540(2) 0.521(2) 0.7336(10) 0.201(17) Uani 1 1 d U . .
H8A H 0.4926 0.5047 0.7277 0.241 Uiso 1 1 calc R . .
H8B H 0.5682 0.5660 0.7229 0.241 Uiso 1 1 calc R . .
C9 C 0.560(3) 0.476(3) 0.7203(13) 0.234(18) Uani 1 1 d U . .
H9A H 0.5284 0.4292 0.7279 0.281 Uiso 1 1 calc R . .
H9B H 0.6059 0.4885 0.7285 0.281 Uiso 1 1 calc R . .
C10 C 0.558(2) 0.484(2) 0.6756(12) 0.232(17) Uani 1 1 d U . .
H10A H 0.5262 0.4984 0.6694 0.348 Uiso 1 1 calc R . .
H10B H 0.5443 0.4400 0.6643 0.348 Uiso 1 1 calc R . .
H10C H 0.6032 0.5167 0.6670 0.348 Uiso 1 1 calc R . .
C11 C 0.5661(13) 0.6393(12) 0.7808(7) 0.124(8) Uani 1 1 d U . .
H11A H 0.5553 0.6374 0.7551 0.149 Uiso 1 1 calc R . .
H11B H 0.5496 0.6669 0.7926 0.149 Uiso 1 1 calc R . .
C12 C 0.6413(14) 0.6753(16) 0.7846(8) 0.149(10) Uani 1 1 d U . .
H12A H 0.6592 0.6509 0.7708 0.179 Uiso 1 1 calc R . .
H12B H 0.6530 0.6751 0.8100 0.179 Uiso 1 1 calc R . .
C13 C 0.674(2) 0.7472(17) 0.7715(11) 0.195(14) Uani 1 1 d U . .
H13A H 0.6601 0.7732 0.7867 0.234 Uiso 1 1 calc R . .
H13B H 0.6597 0.7481 0.7468 0.234 Uiso 1 1 calc R . .
C14 C 0.7548(19) 0.7806(17) 0.7729(11) 0.203(16) Uani 1 1 d U . .
H14A H 0.7679 0.7695 0.7957 0.305 Uiso 1 1 calc R . .
H14B H 0.7758 0.8296 0.7708 0.305 Uiso 1 1 calc R . .
H14C H 0.7697 0.7632 0.7532 0.305 Uiso 1 1 calc R . .
C15 C 0.4536(14) 0.5429(17) 0.7856(11) 0.173(12) Uani 1 1 d U . .
H15A H 0.4517 0.5482 0.7595 0.208 Uiso 1 1 calc R . .
H15B H 0.4387 0.5718 0.7974 0.208 Uiso 1 1 calc R . .
C16 C 0.4042(13) 0.4662(17) 0.7963(9) 0.161(12) Uani 1 1 d DU . .
H16A H 0.3686 0.4452 0.7779 0.193 Uiso 1 1 calc R . .
H16B H 0.4299 0.4422 0.7962 0.193 Uiso 1 1 calc R . .
C17 C 0.371(2) 0.4573(19) 0.8325(12) 0.222(17) Uani 1 1 d DU . .
H17A H 0.3557 0.4899 0.8362 0.266 Uiso 1 1 calc R . .
H17B H 0.4023 0.4616 0.8520 0.266 Uiso 1 1 calc R . .
C18 C 0.3076(19) 0.3792(19) 0.8289(11) 0.26(2) Uani 1 1 d DU . .
H18A H 0.3042 0.3639 0.8042 0.388 Uiso 1 1 calc R . .
H18B H 0.2658 0.3780 0.8356 0.388 Uiso 1 1 calc R . .
H18C H 0.3151 0.3496 0.8447 0.388 Uiso 1 1 calc R . .
C19 C 0.5396(14) 0.5589(14) 0.8338(8) 0.129(7) Uani 1 1 d U . .
H19A H 0.5867 0.5689 0.8356 0.155 Uiso 1 1 calc R . .
H19B H 0.5101 0.5118 0.8417 0.155 Uiso 1 1 calc R . .
C20 C 0.5296(19) 0.6065(15) 0.8594(9) 0.164(11) Uani 1 1 d U . .
H20A H 0.4821 0.5953 0.8582 0.197 Uiso 1 1 calc R . .
H20B H 0.5577 0.6534 0.8509 0.197 Uiso 1 1 calc R . .
C21 C 0.548(2) 0.603(3) 0.8998(10) 0.24(2) Uani 1 1 d U . .
H21A H 0.5280 0.5546 0.9061 0.283 Uiso 1 1 calc R . .
H21B H 0.5976 0.6226 0.9012 0.283 Uiso 1 1 calc R . .
C22 C 0.527(2) 0.637(2) 0.9293(13) 0.26(2) Uani 1 1 d U . .
H22A H 0.5323 0.6789 0.9202 0.386 Uiso 1 1 calc R . .
H22B H 0.5564 0.6461 0.9502 0.386 Uiso 1 1 calc R . .
H22C H 0.4805 0.6065 0.9361 0.386 Uiso 1 1 calc R . .
O1 O 0.6731(5) 0.4647(5) 0.8936(3) 0.060(2) Uani 1 1 d U . .
O2 O 0.6895(6) 0.5737(6) 0.8524(4) 0.082(3) Uani 1 1 d U . .

```
O3 O 0.5983(5) 0.4372(6) 0.8370(3) 0.062(3) Uani 1 1 d U . .
O4 O 0.7872(5) 0.5352(5) 0.8548(4) 0.063(3) Uani 1 1 d U . .
O5 O 0.7109(5) 0.4063(5) 0.8374(3) 0.050(2) Uani 1 1 d U . .
O6 O 0.7120(6) 0.5050(6) 0.7969(4) 0.076(3) Uani 1 1 d U . .
O7 O 0.8185(6) 0.4902(6) 0.7946(4) 0.075(3) Uani 1 1 d U . .
O8 O 0.9140(6) 0.5402(6) 0.8515(4) 0.077(3) Uani 1 1 d U . .
O9 O 0.8084(5) 0.4497(5) 0.8935(3) 0.062(3) Uani 1 1 d U . .
O10 O 0.8259(6) 0.4020(6) 0.9598(3) 0.072(3) Uani 1 1 d U . .
O11 O 0.6994(6) 0.2875(6) 0.9475(3) 0.068(3) Uani 1 1 d U . .
O12 O 0.6667 0.3333 0.8919(5) 0.049(3) Uani 1 3 d SU . .
O13 O 0.7113(8) 0.4114(7) 0.7510(4) 0.093(4) Uani 1 1 d U . .
Si1 Si 0.7571(3) 0.4872(3) 0.76927(19) 0.0771(14) Uani 1 1 d U . .
Si2 Si 0.6667 0.3333 0.7372(3) 0.086(3) Uani 1 3 d SU . .
W1 W 0.69284(4) 0.50107(4) 0.84660(13) 0.0606(3) Uani 1 1 d U . .
W2 W 0.83060(4) 0.48009(4) 0.84506(13) 0.0611(3) Uani 1 1 d U . .
W3 W 0.76686(4) 0.37583(4) 0.92837(13) 0.0579(2) Uani 1 1 d U . .
P1 P 0.6667 0.3333 0.8488(2) 0.0336(12) Uani 1 3 d SU . .
N1 N 0.5247(10) 0.5648(11) 0.7965(7) 0.123(5) Uani 1 1 d U . .

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 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
C1 0.106(17) 0.110(10) 0.091(13) 0.040(10) 0.012(8) 0.036(12)
C2 0.21(3) 0.19(3) 0.20(3) 0.10(2) -0.036(19) 0.09(3)
C3 0.25(3) 0.088(10) 0.15(2) 0.045(12) 0.044(17) 0.025(16)
C4 0.23(3) 0.20(2) 0.18(3) 0.127(19) 0.127(19) 0.13(2)
C5 0.17(2) 0.17(2) 0.058(10) 0.000 0.000 0.083(12)
C7 0.101(16) 0.095(14) 0.172(13) 0.032(15) -0.013(16) 0.046(14)
C8 0.32(5) 0.19(3) 0.178(14) -0.01(3) -0.09(3) 0.19(4)
C9 0.25(4) 0.26(4) 0.25(2) -0.04(3) 0.00(3) 0.17(3)
C10 0.24(4) 0.21(4) 0.25(2) -0.04(3) -0.02(3) 0.12(3)
C11 0.138(12) 0.122(12) 0.15(2) 0.026(13) -0.027(17) 0.091(11)
C12 0.129(12) 0.17(2) 0.17(3) 0.06(2) 0.02(2) 0.091(13)
C13 0.197(19) 0.141(18) 0.19(3) 0.01(2) -0.03(3) 0.042(15)
C14 0.198(19) 0.15(2) 0.21(3) 0.04(2) -0.02(3) 0.049(17)
C15 0.108(12) 0.19(2) 0.26(3) 0.08(2) -0.039(15) 0.097(11)
C16 0.106(15) 0.19(2) 0.19(3) 0.05(2) -0.018(17) 0.076(12)
C17 0.19(3) 0.22(3) 0.24(3) 0.03(3) 0.04(2) 0.09(2)
C18 0.19(3) 0.25(3) 0.26(4) 0.10(3) 0.02(2) 0.05(2)
C19 0.13(2) 0.13(2) 0.160(13) 0.046(13) -0.020(15) 0.086(19)
C20 0.18(3) 0.11(2) 0.186(16) 0.004(19) -0.04(3) 0.07(2)
C21 0.20(4) 0.33(6) 0.146(18) 0.04(3) 0.03(3) 0.12(4)
C22 0.27(4) 0.23(4) 0.20(2) -0.03(3) -0.01(3) 0.08(3)
O1 0.064(6) 0.051(6) 0.080(6) -0.024(4) -0.010(5) 0.040(5)
O2 0.074(8) 0.063(6) 0.118(10) 0.005(6) 0.004(7) 0.041(6)
O3 0.054(5) 0.072(6) 0.079(7) 0.025(6) 0.002(5) 0.045(4)
O4 0.056(5) 0.026(4) 0.103(8) 0.009(5) 0.001(5) 0.017(4)
O5 0.041(4) 0.046(4) 0.069(6) -0.009(4) -0.007(4) 0.026(4)
O6 0.061(6) 0.065(7) 0.097(6) 0.015(5) 0.005(5) 0.029(6)
O7 0.066(6) 0.071(8) 0.090(6) 0.020(5) 0.012(5) 0.035(6)
O8 0.048(5) 0.058(7) 0.115(9) 0.007(6) 0.003(5) 0.020(5)
O9 0.041(5) 0.055(6) 0.082(6) -0.007(5) -0.013(5) 0.019(4)
O10 0.059(6) 0.066(7) 0.088(8) 0.012(6) -0.002(5) 0.029(5)
O11 0.070(7) 0.060(6) 0.077(8) 0.004(5) -0.008(6) 0.034(5)
O12 0.044(5) 0.044(5) 0.058(7) 0.000 0.000 0.022(2)
O13 0.109(10) 0.090(6) 0.069(8) 0.004(5) 0.002(7) 0.040(7)
Si1 0.086(4) 0.070(3) 0.075(3) 0.017(3) 0.008(3) 0.039(3)
Si2 0.097(4) 0.097(4) 0.063(5) 0.000 0.000 0.048(2)
W1 0.0567(5) 0.0453(5) 0.0835(7) 0.0032(4) -0.0029(4) 0.0282(4)
W2 0.0423(4) 0.0472(4) 0.0863(7) 0.0064(4) 0.0028(4) 0.0168(4)
W3 0.0504(4) 0.0545(5) 0.0670(5) -0.0014(4) -0.0080(4) 0.0249(4)
```

```
P1 0.0250(15) 0.0250(15) 0.051(3) 0.000 0.000 0.0125(7)
N1 0.113(11) 0.124(13) 0.167(13) 0.031(12) -0.042(13) 0.084(9)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

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C1 C2 1.43(3) . ?
C1 C3 1.48(4) . ?
C1 C4 1.49(3) . ?
C1 Si1 1.85(2) . ?
C5 C6 1.3000(11) . ?
C5 Si2 1.88(3) . ?
C7 N1 1.51(3) . ?
C7 C8 1.54(4) . ?
C8 C9 1.38(5) . ?
C9 C10 1.65(5) . ?
C11 C12 1.47(3) . ?
C11 N1 1.56(3) . ?
C12 C13 1.48(4) . ?
C13 C14 1.58(5) . ?
C15 N1 1.47(3) . ?
C15 C16 1.56(4) . ?
C16 C17 1.48(5) . ?
C17 C18 1.63(3) . ?
C19 N1 1.43(3) . ?
C19 C20 1.53(4) . ?
C20 C21 1.56(4) . ?
C21 C22 1.53(5) . ?
O1 W1 1.867(12) . ?
O1 W3 1.940(12) 2_655 ?
O2 W1 1.686(12) . ?
O3 W2 1.888(11) 2_655 ?
O3 W1 1.912(11) . ?
O4 W1 1.887(10) . ?
O4 W2 1.955(10) . ?
O5 P1 1.492(10) . ?
O5 W2 2.370(9) . ?
O5 W1 2.387(9) . ?
O6 Si1 1.619(13) . ?
O6 W1 1.869(13) . ?
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O8 W2 1.693(11) . ?
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O10 W3 1.632(12) . ?
O11 W3 1.909(11) 4_665 ?
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O12 P1 1.584(19) . ?
O12 W3 2.373(10) . ?
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Si2 O13 1.609(14) 4_665 ?
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W3 O11 1.909(11) 2_655 ?
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P1 O5 1.492(10) 4_665 ?
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C6 C5 Si2 123(5) . . ?
N1 C7 C8 117(2) . . ?
C9 C8 C7 107(3) . . ?
C8 C9 C10 104(4) . . ?
C12 C11 N1 117.4(19) . . ?
C11 C12 C13 112(3) . . ?
C12 C13 C14 110(3) . . ?
N1 C15 C16 110.8(19) . . ?
C17 C16 C15 114(4) . . ?
C16 C17 C18 101(3) . . ?
N1 C19 C20 114(2) . . ?
C19 C20 C21 114(3) . . ?
C22 C21 C20 119(4) . . ?
W1 O1 W3 153.1(6) . 2_655 ?
W2 O3 W1 149.4(6) 2_655 . ?
W1 O4 W2 122.0(5) . . ?
P1 O5 W2 129.6(6) . . ?
P1 O5 W1 128.6(5) . . ?
W2 O5 W1 89.9(3) . . ?
Si1 O6 W1 139.3(8) . . ?
Si1 O7 W2 135.8(8) . . ?
W2 O9 W3 150.1(6) . . ?
W3 O11 W3 124.2(6) 4_665 . ?
P1 O12 W3 124.3(3) . . ?
P1 O12 W3 124.3(3) . 4_665 ?
W3 O12 W3 91.3(5) . 4_665 ?
P1 O12 W3 124.3(3) . 2_655 ?
W3 O12 W3 91.3(5) . 2_655 ?
W3 O12 W3 91.3(5) 4_665 2_655 ?
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O7 Si1 C1 108.5(9) . . ?
O13 Si2 O13 110.5(5) 4_665 . ?
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O8 W2 O7 102.0(6) . . ?
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O8 W2 O4 99.9(5) . . ?
O9 W2 O4 86.0(5) . . ?
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O7 W2 O4 87.5(5) . . ?
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O9 W3 O1 84.2(4) . 4_665 ?
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O11 W3 O12 72.1(4) . . ?
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O5 P1 O12 106.3(5) 4_665 . ?
O5 P1 O12 106.3(5) . . ?
O5 P1 O12 106.3(5) 2_655 . ?
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